MOP-6823

TITLE: QC CHECKS OF ORS INSTRUMENT PERFORMANCE DURING DATA COLLECTION

SCOPE: Describes the OP-FTIR [OP-FTIR Single Beam Ratio Test, Signal to Noise Test - Noise Equivalent Absorbance (NEA), Saturation of Instrument (Detector Nonlinearity), Random Baseline Noise, and Stray Light] and TDLAS (Instrument Operation Check and Daily Reference Cell Calibration Check) QC and background one-time procedures to be conducted at the field site. In addition, describes the procedures to be conducted on a daily basis for the OP-FTIR [OP-FTIR Single Beam Ratio Test and the Twice Daily QC Procedures on Measurement Data (NEA, Signal Strength, Single-Beam Spectrum, and Wavenumber Shifts and Changes in Resolution), TDLAS (Daily Reference Cell Calibration Check), and Meteorological Station (Daily Reasonableness Checks).

PURPOSE: To provide the procedures and frequency for QC checks for ORS Facility instrumentation to be performed in the field during the field campaign, as well as the potential for corrective action.

1.0 ONE-TIME PROCEDURES AT THE FIELD SITE

These checks are performed once during each field campaign, usually on the first day at the field site. For many of the problems identified by these QC checks, it is possible to take corrective action to improve instrument performance. Some examples of correctable problems include re-alignment of the instrument mirrors to improve signal strength, or manipulating the power source to reduce the amount of electronic noise. Some of the problems identified may not affect data quality, but indicate a potential long-term problem with the instrument which should be corrected by an instrument specialist after the field campaign has been completed. An example of this would be a major degradation of signal strength that could be corrected by re-aligning the internal optics of the instrument. More information on these checks can be found in the US EPA *Compendium Method TO-16: Long-Path Open-Path Fourier Transform Infrared Monitoring of Atmospheric Gases,*[a] and the *ASTM Standard Practice E1982-98* document.[b]

1.1 OP-FTIR

1.1.1 OP-FTIR Single Beam Ratio Test

The Single Beam Ratio Test is a fast and easy check on whether the infrared beam is properly aligned through the Michaelson interferometer. Poor alignment in the interferometer results in considerable cancellation of the high-frequency portion of the infrared spectrum.

Align the instrument on the shortest path length to be used in the field campaign. Collect an interferogram at 8.0 cm^{-1} resolution, with a 10-second sample time. The instrument gain should be set at 1.0. Convert the interferogram to a single beam spectrum. Compare the maximum signal value (Y-axis value) of the spectrum at or near 2000 cm^{-1} to the signal value at or near 4000 cm^{-1}. Record the two signal values and the ratio of the value at 4000 cm^{-1} to the value at 2000 cm^{-1}. The signal value at 4000 cm^{-1} should be greater than 20% of the signal value at the 2000 cm^{-1} wavenumber.

1.1.2 Signal to Noise Test - Noise Equivalent Absorbance (NEA)

The NEA noise is a measure of the instrument noise and is generally used as an instrument quality metric. This test is performed over a very short path to eliminate the contribution of atmospheric sources to the noise. The mirror or bistatic infrared source is placed as close to the OP-FTIR telescope as possible without touching the telescope. This setup is referred to as “Zero Path.” The mirror array should be at least as large as the OP-FTIR primary mirror and set up so that part of the array is in front of every portion of the primary mirror. Set the gain to 1 and collect five one-minute interferograms at 0.5 cm^{-1} resolution (for lower-resolution instruments, set the resolution to the maximum resolution used in the field and expand the three regions (listed below) by a factor of the maximum instrument resolution divided by 0.5. This will ensure that the NEA determination is performed over 80 data points). If analog-to-digital (A/D) overflow or detector saturation occurs, attenuate the beam using either wire meshes or tape strips (to block part of the beam). Create four differential absorbance spectra from the four interferograms by using the first interferogram as the background to process the second interferogram, the second to process the third, the third to process the fourth, and the fourth to process the fifth.

For each of the four differential absorbance spectra, record the root-mean-square (rms) noise in the following three spectral regions:

1. 978 to 998 cm^{-1}
2. 2500 to 2520 cm^{-1}
3. 4390 to 4410 cm^{-1}

Each OP-FTIR sensor will have its own NEA specification. Because different applications may have different performance requirements, there is no specific pass or fail criterion for the NEA test. However, if the NEA determinations in regions 1 or 2 are greater than 0.001 AU, the project leader should be advised of the NEA test results, so that they may determine if the signal-to-noise ratio is sufficient for the intended application. Region 3 is only important if analysis is to be performed on absorption bands at wavenumbers greater than 4000 cm^{-1}. Hydrogen fluoride is generally the only chemical species that is analyzed at wavenumbers higher than 4000 cm^{-1}. The noise specification for Region 3 is a factor of four higher than for the other regions. Poor NEA determinations indicate that maintenance of the instrument, with respect to cleaning and possibly alignment, should be scheduled as soon as possible.

The five Zero-Path interferograms should be saved for possible use in quality testing synthetic backgrounds or to replace synthetic backgrounds as zero-path backgrounds.

1.1.3 Saturation of Instrument (Detector Nonlinearity)

There are two main reasons why the OP-FTIR sensor may respond nonlinearly to changes in the infrared intensity:

1. The electronic gain is set too high, resulting in the A/D converter becoming saturated. When this occurs, the peak signal of the centerburst of the interferogram will be clipped. The adjustment of the electronic gain is set by the pot(s) on the detector preamplifier board. This is only a problem when the A/D is saturated at low signal levels, and where reducing the signal will result in failure of the Signal to Noise Test in Section 1.1.2. In this case, the gain should be reduced by adjusting the pot(s) on the detector preamplifier board, following the manufactures' instructions.
2. There is too much infrared light incident on the detector, resulting in saturation of the detector (the detector no longer provides a linear response to changes in the infrared signal).

To test for detector nonlinearity, set up a mirror at the shortest path length to be used in the study. Align the instrument on the mirror and collect an interferogram for 1 minute at 0.5 cm^{-1} resolution. The software gain of the instrument should be set at 1.0.* Convert the interferogram to a single beam spectrum. Inspect the single beam spectrum in the region below 700 cm^{-1}.** For pathlengths greater than 25 meters, the instrument response should be flat at the zero-signal level. If not, this indicates that the detector is saturated. Block part of the mirror from the infrared beam with a strip of tape and repeat the test. If saturation continues to occur, add another strip of tape and repeat the test. Repeat these steps until detector saturation is eliminated. Then repeat this procedure for the next closest path until detector saturation is eliminated. Continue this procedure with each subsequently longer path until a tested path results in no saturation without using tape. Longer paths need not be tested unless the corresponding peak-to-peak voltage is larger than the shortest path that passed the test.

1.1.4 Random Baseline Noise

To assess the random baseline noise of the system, align the instrument on a mirror to be used in the field campaign. Collect two single beam spectra sequentially at 0.5 cm^{-1} for 10 seconds. The

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* The software gain is the gain that is adjusted in the data acquisition software (with integers from the binary series – 1, 2, 4, 8, etc.) and should not to be confused with the electronic gain.

** For paths shorter than 25 meters (one-way), the response will not be flat. For the shorter paths, the signal strengths should be close to zero at 668 cm^{-1} and 2360 cm^{-1}. If any part of the single beam spectrum has signal values that are negative or less than the values at 668 cm^{-1} and 2360 cm^{-1}, the detector is saturated.

gain of the instrument should be set at 1.0. Analysis of the random baseline noise will be done after the field study is completed.

1.1.5 Stray Light

Some monostatic systems[***] will detect a significant level of the outgoing infrared light that is scattered off of surfaces, inside the instrument enclosure, that are in the detector's field of view. Since this scattered light has not traversed the measured path, it contains none of the absorption bands of the species that are being measured. If the level of the scattered light is more than a few percent of the returned-beam signal, a significant negative bias in the concentration determinations will occur. For instruments operating to specifications, this problem is most apparent in the Midac, occurs to a much lesser extent in the RAM2000, and is generally undetectable in the IMACC. Indeed, the stray light in the IMACC is so low that the center-burst signal is lower than the noise. Under these conditions, collection of stray-light interferograms is not possible.

The test for stray light is a two-step process in which the first step will determine if any stray light is detected. If the first step detects stray light, then the second step will quantify the stray-light level and provide, if necessary, a relatively low-noise spectrum to correct the measured spectra for stray light. Once the stray light intensity is known and measured, it should not change unless some component of the optical system is changed.

1. In alignment mode, align the beam to a mirror. After observing a definite return interferogram signal, slew the OP-FTIR far from the mirror and observe if an interferogram signal is detected. If the signal disappears, the instrument does not experience stray light and no further test will be required. If an interferogram is detected, go to step 2.
2. While the instrument is still slewed away from the mirror, set the resolution to 8.0 cm^{-1} and the gain to a value that maximizes the peak-to-peak signal without A/D overflow. Collect 16 one-minute samples. Convert the 16 interferograms to 16 single-beam spectra. Co-add the 16 one-minute single-beam spectra to produce one 16-minute averaged single beam.

   During the data analysis phase of the field project, the signal level of the co-added 16-minute single-beam stray light spectrum will be compared to the level of the measurements at 1000 cm^{-1} and recorded for the report. If the stray light level is significant (> 5 percent), the measurement results can be corrected by one of two different methods:

   a. The 16-minute single-beam spectrum is interpolated to match the data points of the measured spectra. The interpolated stray-light single beam is then subtracted from the single-beams that were obtained by performing a Fourier transform of the measurement interferograms. Quantitative analysis is then performed on the resulting processed single beam spectra.

*** Bistatic systems do not experience stray light, but they are subjected to ambient radiation, which has a similar effect on the concentration measurements.

b. The field-measurement spectra are first analyzed to obtain concentrations. The signal-strength of the uncorrected single beams is listed along with the corresponding peak absorptions and concentrations. The concentrations are corrected for the stray light using

   information on the stray light single-beam signal and the returned beam signal at 1000 cm^{-1}, and the peak absorption of the measured chemical absorption band.

The second method may be preferred if the first method produces too much noise. If the corrections are too labor intensive, more approximate correction procedures may be used. A reliable estimate of propagated error should be reported along with approximate corrections.

1.2 OP-TDLAS

1.2.1 Instrument Operation Check

Once in the field, the first QC check necessary is ensuring that the instrument is operational. After the instrument is turned on, absorption peaks should become visible on the screen. No warm-up period is required. There are two absorption peaks visible in the software, sample and reference. The sample peak is only visible when the gas species selected is present within the open path. The reference peak is visible all the time, even without a telescope connected. This is a commercial piece of equipment intended for data collection. The data stored is in path average concentrations (ppm) identified by the sample gas and telescope selected.

1.2.2 Daily Calibration Check

At the beginning of the field campaign, a single reference cell will be used to check the calibration for each analyte being investigated. Refer to MOP-6802, Section 1.4.2, for further information on the calibration check procedure.

The instrument response for the reference cell of each analyte of interest is evaluated. For the instrument to be considered functioning within operational parameters the accuracy must fall within +/- 10% of the known value, and the precision of duplicate measurements must be within +/- 5%.

2.0 PROCEDURES TO BE CONDUCTED ON A DAILY BASIS

2.1 OP-FTIR

2.1.1 OP-FTIR Single Beam Ratio Test

The OP-FTIR Single Beam Ratio Test (described in Section 1.1.1) should be performed once, at the beginning of each day of the field campaign, to document that the instrument is continuing to operate within acceptable QC limits.

2.1.2 Twice Daily QC Procedures on Measurement Data

The document *ASTM Standard Practice E1982-98* lists five procedures that are recommended to be conducted twice a day. However, these tests can be performed after the study has concluded, on measurement data collected from two different times during each day of the sampling event. By making the responsibility of following these procedures a part of the post-measurement analysis phase, greater productivity and cost effectiveness will be achieved during the field campaign, without compromising data quality. Each of the recommended QC checks will be discussed below.

2.1.2.1 Noise Equivalent Absorbance (NEA)

The measurement portion of this procedure does not need to be performed separately from the program measurements, and the procedure itself may be followed during a post-measurement period. The twice-daily requirement can be satisfied by performing the analytical procedure on two consecutive measurements made at the start and at the end of each day of data collection.

Choose two representative, sequential interferograms collected over one single path from the measurement data set that was collected within the first hour of the day's measurement campaign. Create an absorption spectrum from these two spectra by using one spectrum as a background spectrum. Which spectrum is used for the background is not important. Measure the random noise as the root-mean-square (RMS) noise. The actual wavenumber range over which the noise should be calculated will vary with the number of data points per wavenumber in the spectrum. A range of at least 80 data points should be used for the RMS noise calculation. The RMS noise should be determined in wavenumber regions that are not significantly impacted by water vapor; for example, 958–1008 cm^{-1}, 2480–2530 cm^{-1}, and 4375–4425 cm^{-1}. Record the value of the RMS noise for future reference.

Repeat this procedure on two representative, sequential spectra collected over one single path within the final hour of the day's measurement campaign. This procedure should be repeated for each day of the field campaign.

2.1.2.2 Signal Strength

This procedure can also be performed as part of the post-measurement analysis. Choose a representative interferogram that was measured within the first hour of the day's measurement campaign. Measure the signal strength as the peak-to-peak voltage of the interferogram centerburst and record the position of the zero peak difference. Note the distance to the mirror array and if the size or condition of the array may have an affect on the NEA value. Note also any unusual atmospheric conditions, such as fog, snow, or heavy rain that might affect the signal strength.

Repeat this procedure on a representative interferogram that was collected within the final hour of the day's measurement campaign. This procedure should be repeated for each day of the field campaign.

2.1.2.3 Single-Beam Spectrum

This procedure can also be performed as part of the post-measurement analysis. Choose a representative interferogram that was measured within the first hour of the day's measurement campaign. Convert the interferogram to a single-beam spectrum. Examine the single-beam spectrum for evidence of nonlinear response. Measure the single-beam intensity in different wavenumber regions; for example, near 990, 2500, and 4400 cm^{-1}, to determine if the output power of the IR source, the transmitting or reflecting properties of the optics, or the alignment of the interferometer have changed.

Repeat this procedure on a representative interferogram that was collected within the final hour of the day's measurement campaign. This procedure should be repeated for each day of the field campaign.

2.1.2.4 Wavenumber Shifts and Changes in Resolution

The following post-measurement procedure involves manual manipulation of absorbance spectra. The results from this procedure are not quantitative and require some interpretation. Small shifts and changes in resolution might not produce the derivative- and W-shapes (described below). A software algorithm can be written to automatically measure and compare the wavenumber shifts and changes in resolution to measure the line center and linewidths of selected water vapor lines. By comparing the line centers and the line widths of two spectra, one can make a quantitative determination of the wavenumber shift and the change in resolution. This algorithm will reduce the effort that is required by this task and will provide quantitative results.

Meanwhile, the line centers can be determined directly using OMNIC. Choose from the "Edit" Menu, "Options", then select the "View" tab. In the "Annotation" box, enter "3" in the selection for number of decimal places. Isolate the selected water vapor line in the window (box the line so that other stronger lines do not appear) and choose "Find Peaks" in the "Analyze" Menu. The line center measurement value will appear above the line with 0.001 cm^{-1} precision.

This procedure can also be performed as part of the post-measurement analysis. Conduct the following tests to determine if wavenumber shifts or changes in resolution have occurred during the acquisition of each data set. These tests should also be conducted whenever the OP-FTIR monitor has been moved to change the path; optical components in the system have been changed or realigned; or the instrument has been disassembled, shipped, and reassembled.

1. Select absorption spectra taken at different times during the study; for example, near the beginning, middle, and end of the study.
2. Compare the peak maxima in absorbance of selected narrow water-vapor lines to determine visually if a change has taken place between the spectra in the data set.
3. Subtract one absorption spectrum from the other. The bands in the two spectra being subtracted must be of the same intensity, or they must be scaled to the same intensity prior to the subtraction operation.

4. If a wavenumber shift has occurred between the two spectra, the subtraction result will exhibit a feature that appears to be the first derivative of the band shape.
5. If a change in resolution has occurred, but there is no wavenumber shift, the subtraction result will exhibit a feature that has the shape of an *M* or a *W*, depending on which of the two spectra contains the broader band.

6. If there are no wavenumber shifts or changes in resolution, the result of subtraction will be random noise.

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2.3 The Meteorological Station

2.3.1 Daily Reasonableness Checks

A couple of reasonableness checks must be performed daily on the measured wind direction data. While data collection is occurring, the measured wind direction should be compared to the forecasted wind direction for that particular day. Additionally, the observed wind directions measured with the two heads (if two meteorological stations are deployed) should be compared on a daily basis. If the difference between the two measured wind directions is greater than 60º, data collection should be stopped and the instruments should be checked to ensure they are operating correctly.

3.0 REFERENCES

a. U.S. Environmental Protection Agency, *Compendium Method TO-16: Long-Path Open-Path Fourier Transform Infrared Monitoring of Atmospheric Gases,* prepared under Contract No. 68-C3-0315, WA No. 3-10, Center for Environmental Research Information-Office of Research and Development, US EPA, Cincinnati, Ohio, Jan. 1999.

b. *American Society for Testing and Materials (ASTM) Standard Practice E1982-98, Standard Practice for Open-Path Fourier Transform Infrared (OP/FT-IR) Monitoring of Gases and Vapors in Air*, March, 1999.

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